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TENSILE PROPERTIES OF TUNGSTEN 3% RHENIUM FROM 1400 TO 2900°C IN VACUUM*

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Abstract

The tensile properties of recrystallized, doped powder-metallurgy tungsten-3% rhenium rod have been determined from 1370 to 2930°C in vacuum at a strain rate of 0.02/min. A comparison is made with recrystallized, doped powder-metallurgy tungsten rod, which shows comparatively poor ductility, and plasma-flame single-crystal tungsten, a very ductile material. The variation of strain-hardening exponent with temperature is shown for the two powder metallurgy materials. Tungsten-3% rhenium has the highest strength and the lowest ductility of the three materials. At a test temperature of ≃50% of the absolute melting point, both tungsten-3% rhenium and powder-metallurgy tungsten show decreased ductility and intercrystalline fracture associated with void formation and growth. Ductility does not increase with increasing temperature above ≃65% of the absolute melting point for tungsten-3% rhenium, as it does for powder-metallurgy tungsten, and this is attributed to the absence of strain-induced grain growth during testing.

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Unc [redacted]

Introduction

The high melting point, high elastic modulus, and low vapor pressure of rhenium suggest its use as a favorable alloying element for tungsten. Geach and Hughes[1] showed that tungsten-rhenium alloys were far more ductile than the component metals and that a tungsten-35% rhenium alloy was fabricable without difficulty at a few hundred degrees Centigrade. Other work[2,3,4] confirmed and extended the knowledge of the beneficial effects of rhenium on the workability of tungsten in the range from 18 to 30 wt. %. Pugh et al[5], investigating tungsten-rhenium (1–20%) wire for electronic and lamp applications, showed that it has better ductility, higher recrystallization temperature, higher strength, and higher electrical resistance than unalloyed tungsten wire. Tensile properties reported have been for highly wrought forms of tungsten-rhenium alloys, such as wire and sheet, up to about 2000°C. Creep-rupture properties for tungsten-25% rhenium sheet up to 2600°C are reported[6]. To the author's knowledge, tensile properties of dilute tungsten-rhenium alloys in wrought recrystallized coarse-grained rod form at temperatures up to 2900°C have not previously been investigated.

The purpose of the present investigation was to study the possible beneficial effects of 3% rhenium on the tensile properties of doped powder-metallurgy tungsten in swaged, recrystallized rod form. To show contrasting tensile behavior, the data from a previous study[7] on two other tungsten materials are included.

Materials

The swaged tungsten-3% rhenium (W-3Re) rod 0.330 in. in dia. used in this investigation was made by a powder-metallurgy process. It was the same rod stock used to make wire for the study reported in Ref. 5. The powder-metallurgy (PM) tungsten rod, 0.375 in. in dia. and the W-3Re rod were made from doped powder stock, although not from the same lot. Plasma-flame single-crystal (PF) tungsten rods, 0.875 in. in dia., were grown, as the name implies, by melting powder in a plasma-flame hot zone. The crystal axes were parallel to [111] within $\pm$ 10°.

The impurity levels (determined by a commercial laboratory) for W-3Re, PM, and PF tungsten materials are given in Table I. The W-3Re alloy contains more oxygen and carbon and less nitrogen than PM tungsten. Notably high carbon content sets the PF tungsten apart from the other materials. Silicon, iron and aluminum levels are lower for W-3Re than for PM tungsten. Duplicate samples were not run for the W-3Re, hence a range of values is not given. The analysis of typical powder-metallurgy wire is presented for comparison.

Specimens of all three materials were heated, several at once, in vacuum (10^{-5} torr or better) to 2645°C, held for 10 minutes, and furnace cooled prior to testing. This heat treatment resulted in fully recrystallized structures for both W-3Re and PM materials, while only a few low-angle boundaries were observed in the PF tungsten. The "as received" and "as recrystallized" longitudinal microstructures for W-3Re are shown in Figs. 1 and 2, respectively. The structure in Fig. 1 appears to be incompletely recrystallized, while Fig. 2 shows complete recrystallization with secondary grain growth. Unresolved detail within the grains in Fig. 2 occurs with greater frequency than in recrystallized PM tungsten (not shown). The recrystallized grain size for the W-3Re alloy, determined by the line intercept method, varied between 100 and 260 grains/mm^2, with an average of 165 grains/mm^2. The PM material had an average recrystallized grain size of 240 grains/mm^2.

Apparatus

The apparatus and procedure have been described[8]. Briefly, the method involves a vacuum atmosphere and radiation heating of a standard specimen (0.640 in. gage length by 0.160 in. dia.)[9] held in a hot-grip assembly. The furnace is heated by induction. The simultaneous recording, by an x-y plotter, of load cell (strain-gage type) and extensometer (linear potentiometer type) outputs gives an engineering stress-strain curve for each test. All tests were conducted at a strain rate of 0.02/min. Two improvements have been made since the apparatus was first reported[8], namely, a ball nut lead screw has replaced the Acme thread lead screw and a new vacuum system has been installed, giving higher pumping capacity and lower ultimate pressure ($\simeq 5 \times 10^{-6}$ torr) at test temperatures.

Results and Discussion

Stress-Strain Curves and Strain-Hardening Exponent

A typical stress-strain curve at 1900°C and a strain rate of 0.02/min. is replotted from the original x-y record for tungsten-3% rhenium (W-3Re) in Fig. 3. The powder metallurgy (PM) and plasma-flame single-crystal (PF) tungsten curves[7] at the same strain rate and temperature are given for comparison. It is apparent that W-3Re, although stronger, has less elongation than PM tungsten. Both W-3Re alloy and PM tungsten specimens fracture in an intercrystalline manner at 1930°C with little or no necking, while PF material characteristically shows a knife-edge fracture.

Numerical values from the original x-y records were punched on tape for computer handling. The computer was programmed to replot the engineering stress-strain curve, and, assuming constant volume in the specimen during straining, to give the true-stress-true-strain curve. The assumption of constant volume is not quite true because of void formation and growth during the test. The void content expressed on an area basis was 3.9% in a longitudinal section slightly away from the fracture in the W-3Re specimen tested at 1930°C. Further programming produced the logarithm of the true-stress vs. the logarithm of the true-strain curve retraced in Fig. 4.

The simplest mathematical expression for a true-stress-true-strain curve is

$$\sigma = K\epsilon^n$$

where σ is the true stress, K is the strength coefficient, ϵ is the true strain, and n is the strain-hardening exponent. The slope of the line in the Naperian log-log plot (Fig. 4) gives the value for n. The nearly straight line shown up to the maximum true stress indicates that the above mathematical expression fits the data. The rapid drop in true-stress values is probably the result of internal necking, because pores were observed in the fracture zone. As previously mentioned, fracture always occurs in an intercrystalline manner, with little or no external necking, in both the W-3Re alloy and powder-metallurgy (PM) tungsten.

The strain-hardening exponent values for W-3Re are shown in Fig. 5 along with the average curve* for PM tungsten. These data show that a 3% addition of rhenium lowers the strain-hardening exponent for doped powder metallurgy (PM) tungsten between 1370°C and ≃2250°C and above ≃2600°C. The anomaly at ≃1900°C, apparent in Fig. 5 for PM tungsten, has been found by the author (unpublished work) in two other powder-metallurgy materials, one doped and one undoped. The reason for the anomaly, or its absence in the case of W-3Re, is not presently understood.

No obvious relation appears to exist between solid-solution softening or hardening at room temperature and strain-hardening at high temperatures. Rhenium additions up to 5% lower the room temperature hardness level of tungsten[4,5]. In the present work the 500 gram Knoop hardness of W-3Re "as received" was 478 at room temperature. When recrystallised, the hardness fell to 355 Knoop. The recrystallised PM material had an intermediate hardness value at 400 Knoop, substantiating Refs. 4 and 5.

<u>Tensile Data</u>

The tensile data are summarised in Figs. 6–8. Average curves from the data of Ref. 7 are included. Figure 6 shows the ultimate tensile strength of W-3Re as a function of temperature. The W-3Re material exhibits greater strength than either comparison material at all test temperatures except at 2930°C where it has equal strength with PM tungsten. As might be expected, the PF tungsten, even with its higher carbon content, is weaker at all test temperatures.

*A corrected table for the curve of Ref. 7 appears in Trans. ASM, 56 (1963) 973

In Fig. 7 elongation is plotted versus temperature for W-3Re. It is the second form of tungsten which the author has found to show less elongation than doped powder metallurgy rod, the first being pyrolytic tungsten[10].

Ductility differences are even more noteworthy in Fig 8, which shows reduction in area as a function of temperature. The curve for W-3Re lies below that for PM tungsten at all temperatures and does not show any increase in ductility with increasing temperature. Above 1900°C the reduction-in-area values for W-3Re fall below those for pyrolytic tungsten[10] (not shown). The very ductile PF material (knife-edge fractures) shows 99 % reduction-in-area values across the plot.

The photomicrograph in Fig. 9 shows a longitudinal section of a W-3Re specimen tested at 2480°C. Voids lie in general on grain boundaries which are normal to the tensile axis. The number of voids increases with increasing temperature, and the amount of plastic deformation of the grains decreases with increasing temperature — all consistent with the low reduction-in-area and elongation values shown.

One may well ask why the expected beneficial effect of rhenium on ductility when alloyed with tungsten is not realized in this case. The remarkable strength and ductility of W-1Re, W-3Re, and W-5Re in the form of 0.008 in. wire was strongly structure dependent as reported by Pugh[5]. For example, the strongest alloys had the highest recrystallization temperature and when tested at ≃ 2000°C were still very fine-grained, probably only stress-relieved, structures. The structure of the present material is very different, being fully recrystallized and coarser-grained. No direct comparison of the present material can be made with fine wires or thin sheets, even when recrystallized, because of the specimen size factor. A meaningful comparison is possible between the W-3Re rod and PM rod because they were made from the same type of doped tungsten powder stock and had similar pressing and sintering and nearly identical recrystallization and testing conditions.

In the PM material, the ductility minimum, associated with intercrystalline fracture, appears to be the result of void nucleation and growth due to grain boundary sliding[11] at $\approx 0.5\ T_m$. (T_m is the melting point, °K.) An increase in ductility[7], as measured by either reduction in area or elongation, is accompanied by strain-induced grain growth during testing, beginning at ≈ 2100°C ($0.65\ T_m$). The W-3Re alloy showed no increase in ductility in the temperature range above ≈ 2100°C and little or no increase in grain size, with the exception of the specimen* tested at ≈ 2500°C. In pyrolytic tungsten the lack of an appreciable increase in ductility above ≈ 2000°C was also associated with little or no strain-induced grain growth[10]. This is supporting evidence for the dependence of ductility on strain-induced grain growth in tungsten above the grain boundary sliding region at ≈ 0.5 to $\approx 0.65\ T_m$.

Strain-induced grain growth, as well as grain boundary sliding, seems quite dependent upon impurity level and distribution and probably upon grain size. One of the principal effects of rhenium in tungsten is reported[2] to be the lowering of interstitial element solubilities. The W-3Re alloy, with its lower solubility and higher oxygen and carbon content, could have a second phase at the grain boundaries, probably an oxide[2,4]. Second-phase particles, if present, would act to pin grain boundaries and account

*This specimen had the finest grain size, namely 260 grains/mm^2.

for higher strength and the absence of strain-induced grain growth. Solid-solution strengthening may also, in part, account for the higher strength. For example, it has been shown[2,12] in tungsten-rhenium alloys that the grain boundary is rhenium rich and hence harder than the center of the grain.

In the present work the grain size difference (165 grains/mm^2 for W-3Re vs. 240 grains/mm^2 for PM tungsten) is not considered to contribute to the difference in reduction-in-area ductility. In the case of PF tungsten, the absence of grain boundaries and hence a grain boundary distribution of impurities probably accounts for the lower ultimate strength and higher ductility. The effect of crystal orientation is not known.

The limited quantity of W-3Re rod necessitated the use of one test specimen per temperature (with the exception at 1370°C). The quantity limitation was also true for the PF tungsten. From previous work[7] and present work (unpublished), it is felt that the smooth curves shown are sufficiently accurate for the conclusions drawn. The fact that the powder-metallurgy and plasma-flame single-crystal tungsten were tested before the apparatus was improved is not considered significant. It is important to realize that properties reported may belong uniquely with the material lot and that the next lot could have differing properties

Conclusions

1. Doped powder-metallurgy tungsten containing 3% rhenium has higher ultimate strength than the same type of tungsten without rhenium (both in swaged, recrystallized rod form) in the temperature range between 1370 and 2930°C. Three percent rhenium lowers the room temperature hardness of tungsten as recrystallized.

2. Ductility, above 1370°C, as measured by reduction in area and elongation, is lowered by the addition of 3% rhenium to doped powder-metallurgy tungsten. The reduction-in-area ductility above $\simeq$ 2000°C is slightly lower than that reported for pyrolytic tungsten.

3. Decreasing ductility in both powder metallurgy materials is associated with void formation and growth and intercrystalline-type fracture in the temperature range from $\simeq$ 0.5 to $\simeq$ 0.65 T_m (1650 - 2200°C). Strain-induced grain growth, which may limit void growth, appears to be necessary for an increase in ductility above this region.

4. The strain-hardening exponent values above $\simeq$ 1500°C are lower for W-3Re than for powder-metallurgy tungsten without rhenium.

Acknowledgment

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FIGURE CAPTIONS

1. Tungsten-3% rhenium, longitudinal section, "as received". Etched 1 min. in 30cm^3 lactic acid, 10cm^3 HNO_3, and 10cm^3 HF. (100 x)
2. Tungsten-3% rhenium, longitudinal section, "as recrystallized" (10 min. at 2845°C). Etched 1 min. in 30cm^3 lactic acid, 10cm^3 HNO_3, and 10cm^3 HF. (100 x)
3. Typical engineering stress-strain curve for tungsten-3% rhenium tested at 1930°C at a strain rate of 0.02/min. Comparison data from Ref. 7.
4. Naperian log-log plot of true-stress versus true-strain for tungsten-3% rhenium tested at 1930°C at a strain rate of 0.02/min.
5. Strain-hardening exponent n as a function of temperature for tungsten-3% rhenium alloy. Comparison data from Ref. 7.
6. Ultimate tensile strength as a function of temperature for tungsten-3% rhenium alloy. Comparison data from Ref. 7.
7. Elongation as a function of temperature for tungsten-3% rhenium alloy. Comparison data from Ref. 7.
8. Reduction in area as a function of temperature for tungsten-3% rhenium alloy. Comparison data from Ref. 7.
9. Tungsten-3% rhenium, longitudinal section as tested at 2480°C at a strain rate of 0.02/min. Etched 1 min. in 30cm^3 lactic acid, 10cm^3 HNO_3, and 10cm^3 HF. (100 x)

LIST OF TABLES

TABLE I

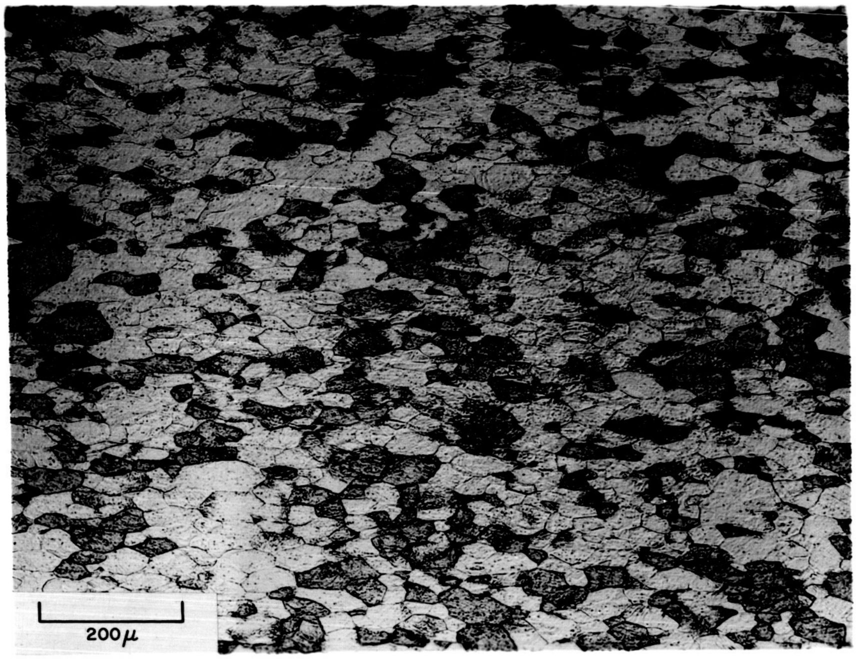
IMPURITY LEVELS IN TUNGSTEN MATERIALS

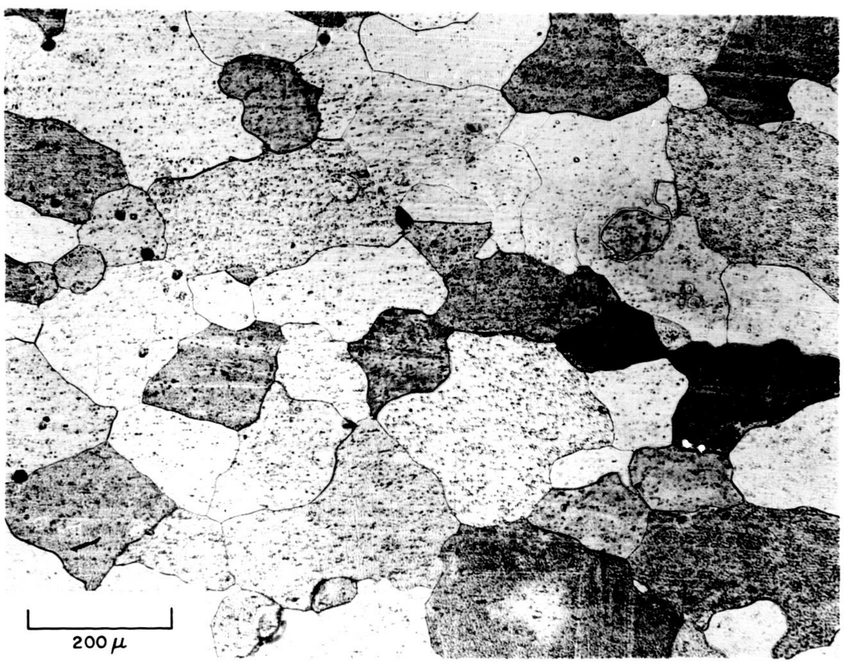
Element	Concentration (p.p.m.)			
		Tungsten		
	W-3Re*	Wire**	Powder Metallurgy	Plasma Flame
Oxygen	28	5	1.3-5	5-9
Carbon	14	20	2-4	66-98
Nitrogen	6	3	5-16	4-7
Hydrogen	< 1	***	***	***
Silicon	< 10	< 10	< 10-20	10-30
Iron	< 10	60	10-40	20-40
Molybdenum	< 50	30	30-50	***
Aluminum	< 10	< 10	20-40	10-30
Nickel	< 10	< 10	< 1	< 10

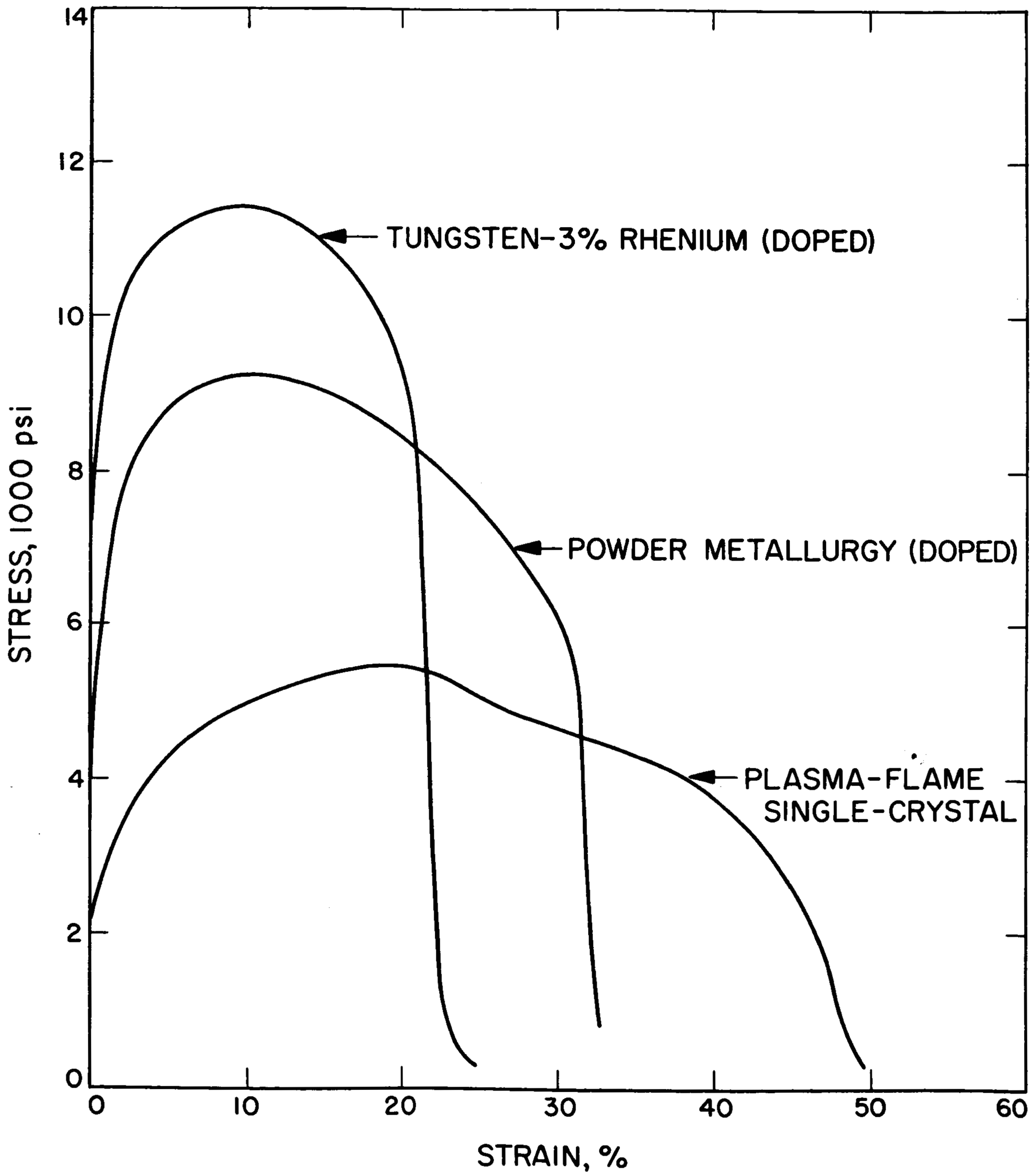
*Actual rhenium content is 2.8% (Ref. 5).

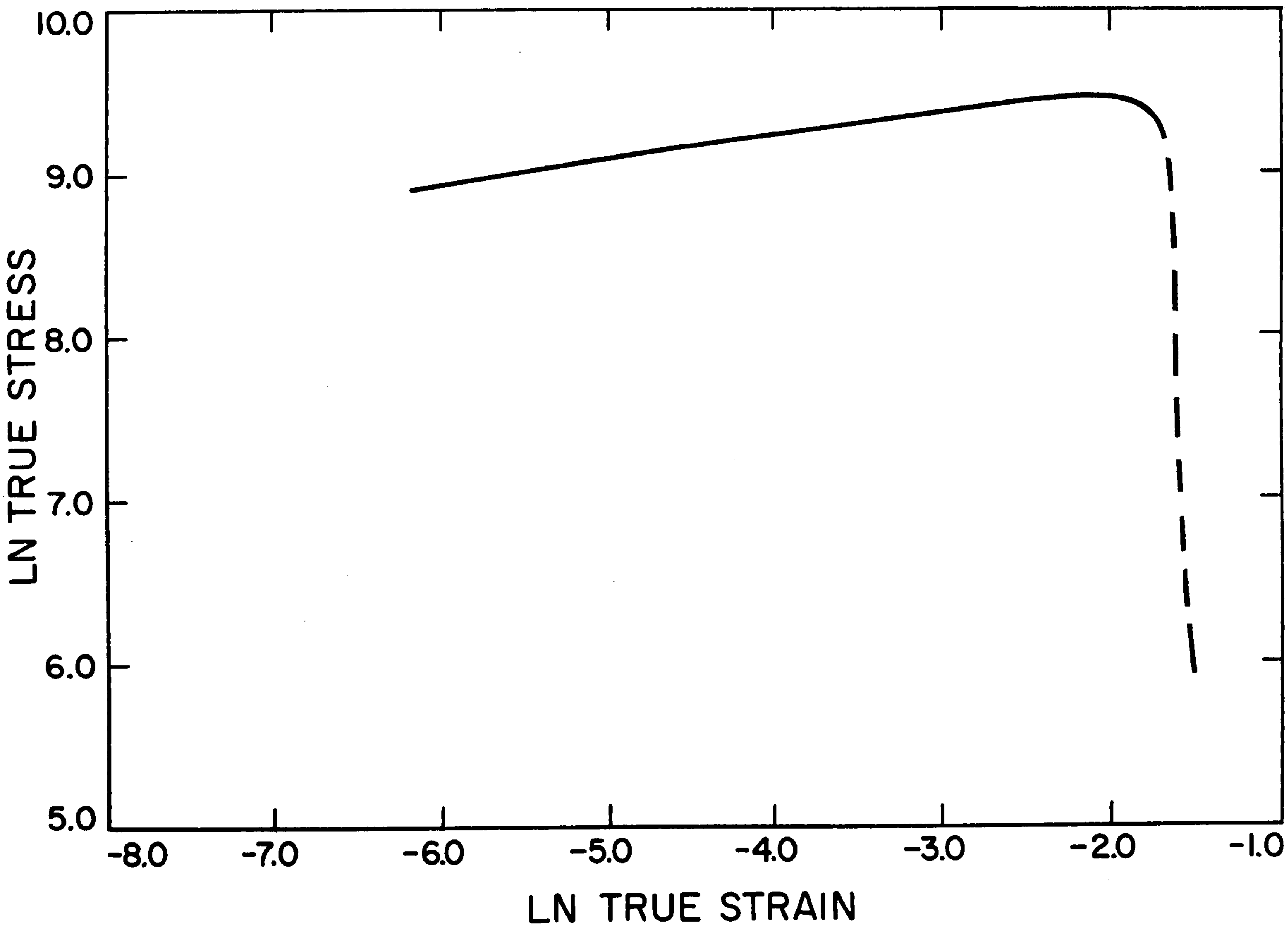
**Typical analysis of tungsten wire (Ref. 5).

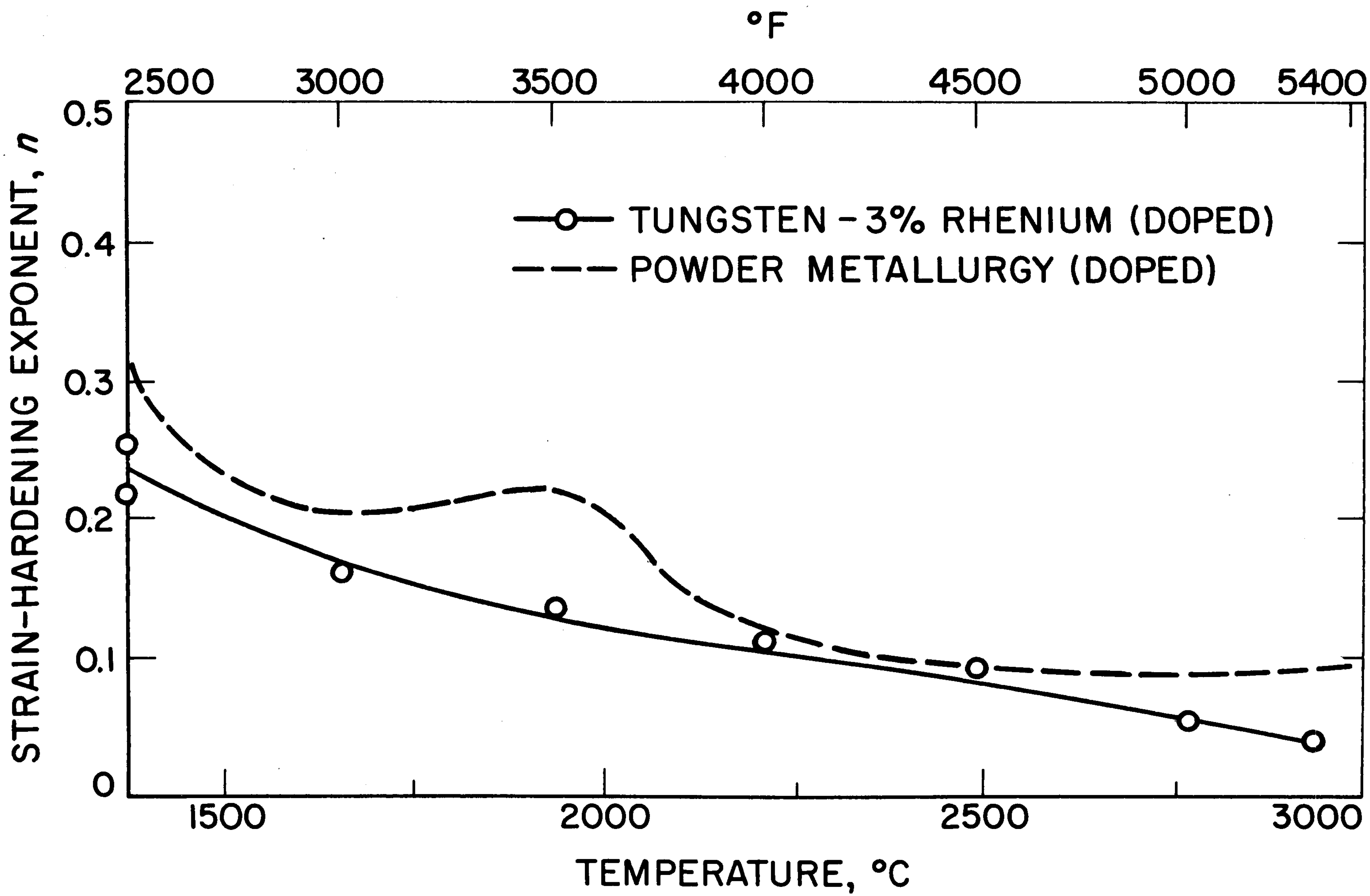
*** Not Determined.

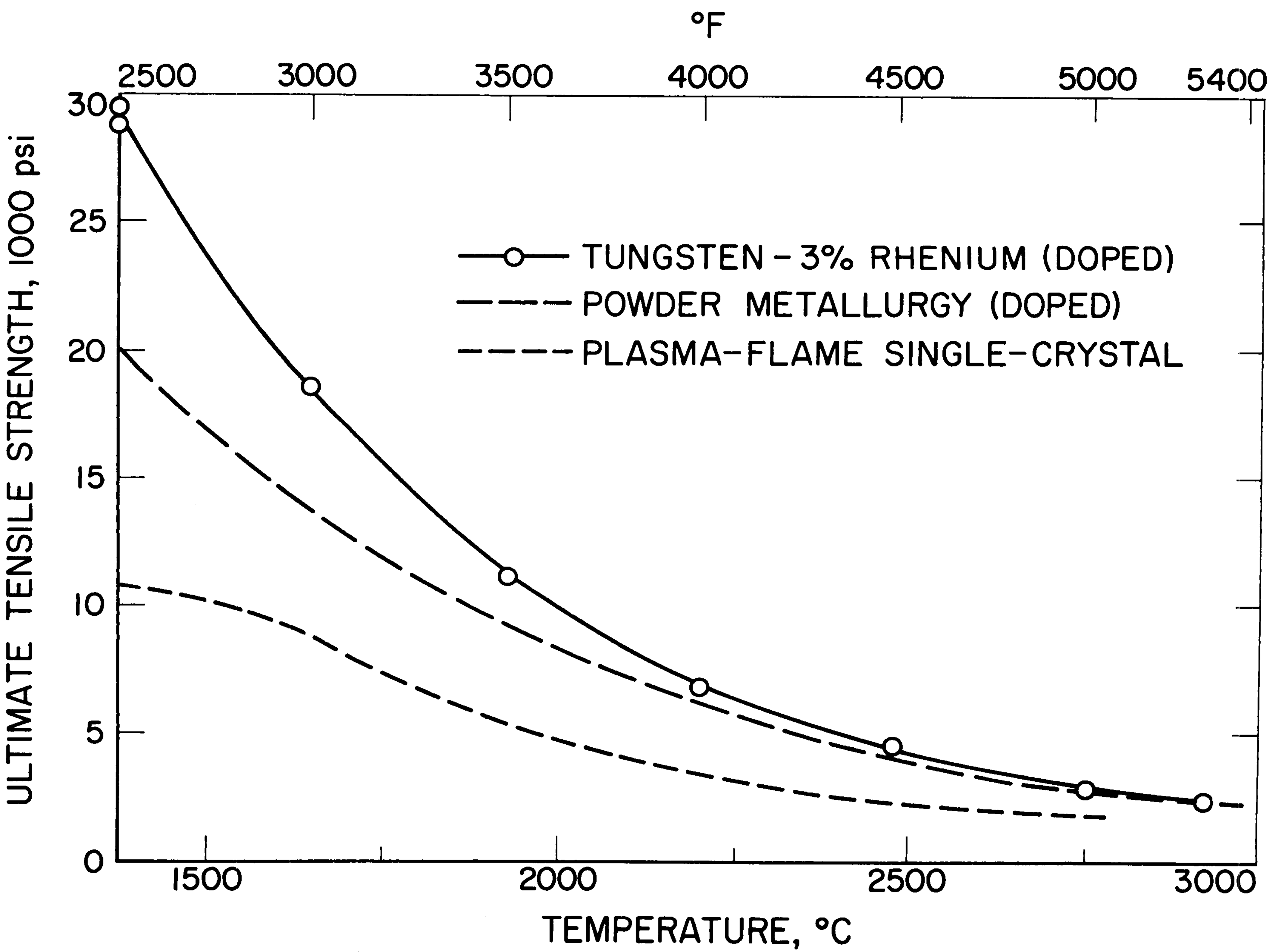

200 μ

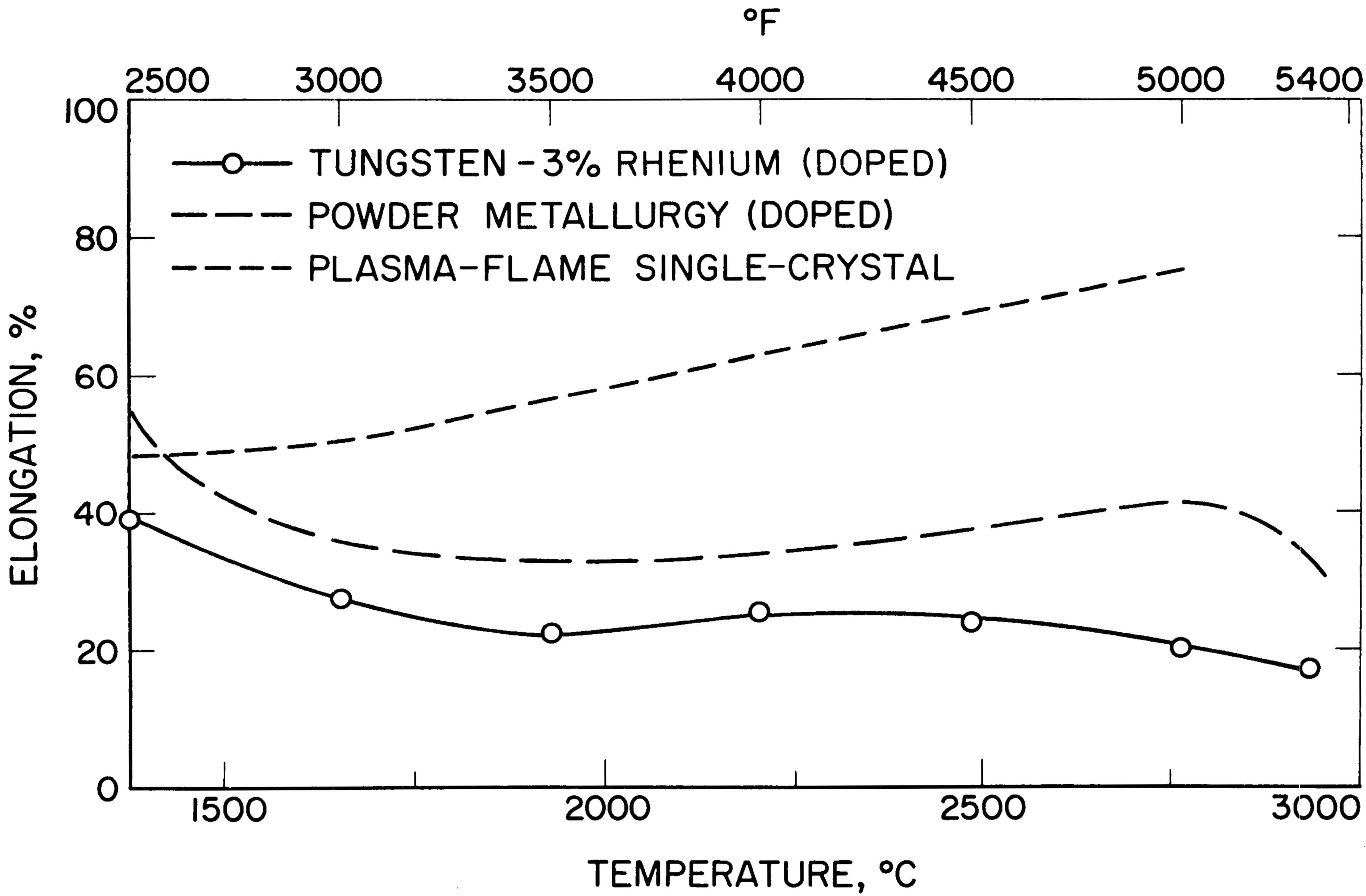

200 μ

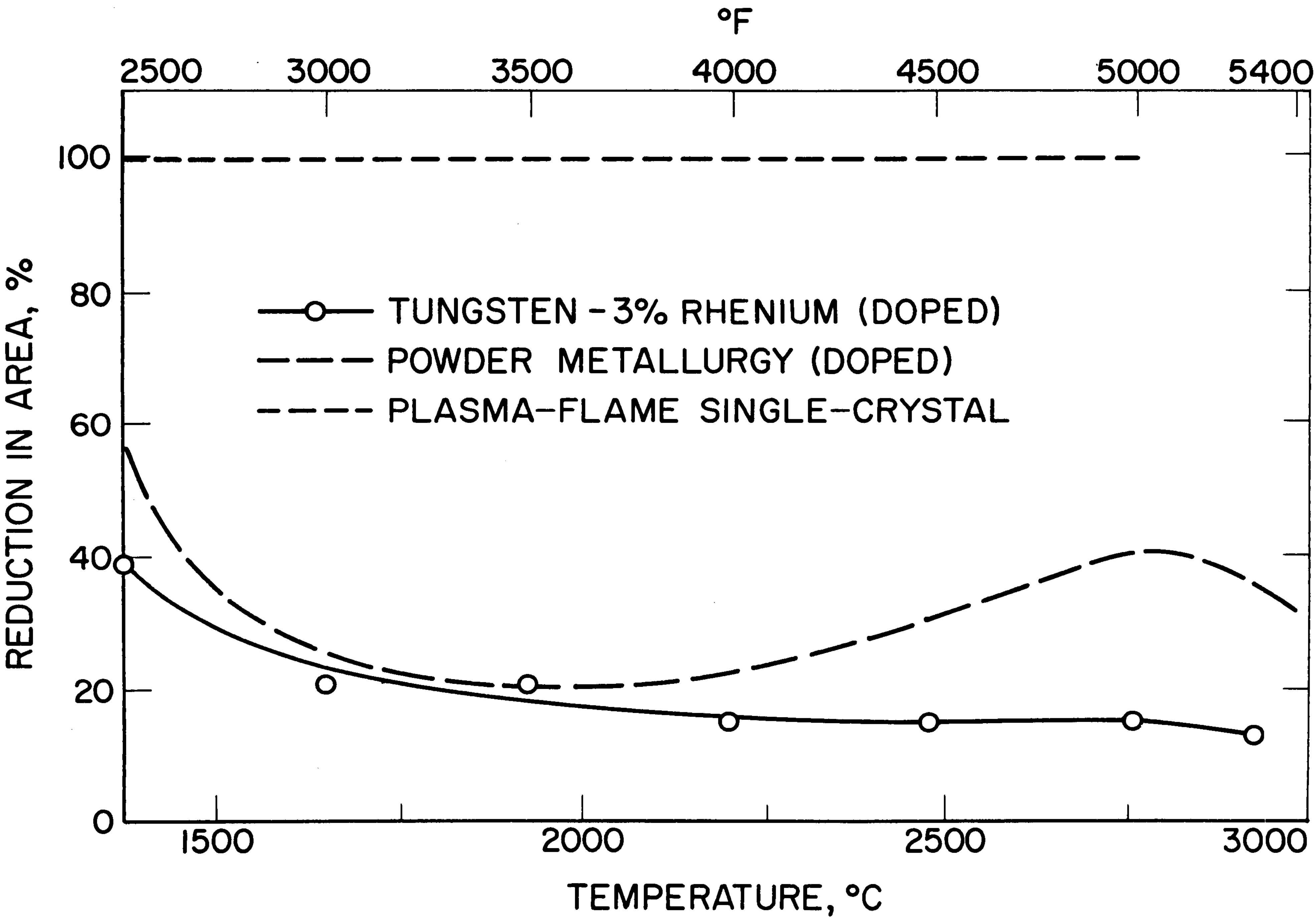

STRESS, 1000 psi
14
12
10
8
6
4
2
0
0
10
20
30
40
50
60
STRAIN, %
TUNGSTEN-3% RHENIUM (DOPED)
POWDER METALLURGY (DOPED)
PLASMA-FLAME
SINGLE-CRYSTAL

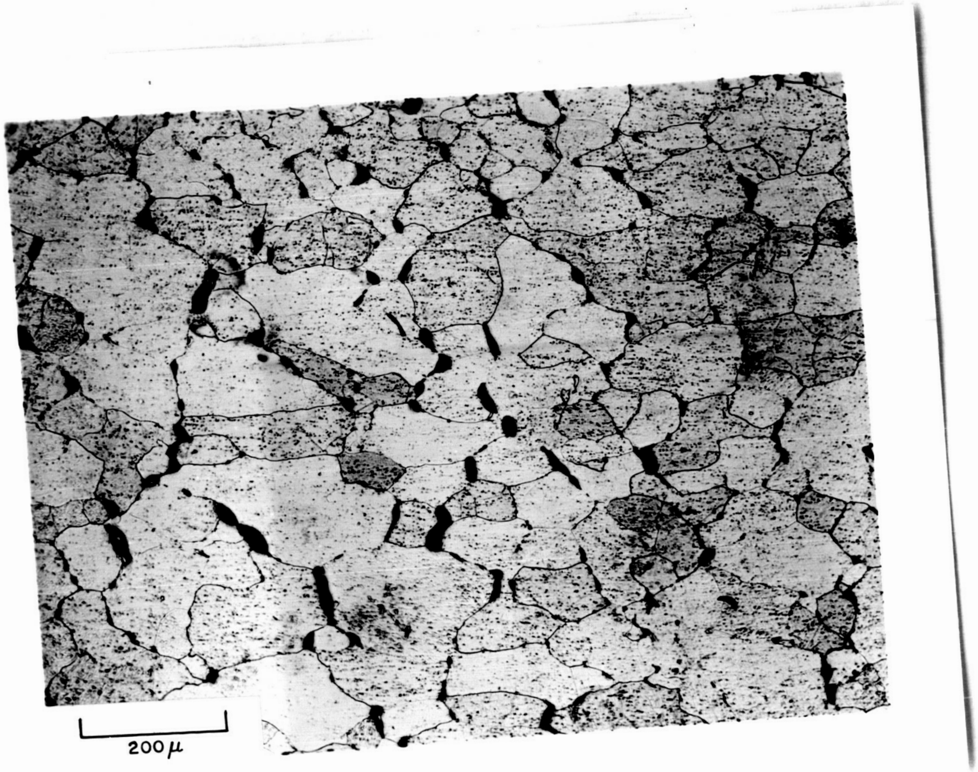

10.0
9.0
8.0
7.0
6.0
5.0
LN TRUE STRESS
-8.0
-7.0
-6.0
-5.0
-4.0
-3.0
-2.0
-1.0
LN TRUE STRAIN


°F
2500
3000
3500
4000
4500
5000
5400
TUNGSTEN - 3% RHENIUM (DOPED)
POWDER METALLURGY (DOPED)
STRAIN-HARDENING EXPONENT, n
0.5
0.4
0.3
0.2
0.1
0
1500
2000
2500
3000
TEMPERATURE, °C


°F
2500
3000
3500
4000
4500
5000
5400
ULTIMATE TENSILE STRENGTH, 1000 psi
0
5
10
15
20
25
30
TUNGSTEN - 3% RHENIUM (DOPED)
POWDER METALLURGY (DOPED)
PLASMA-FLAME SINGLE-CRYSTAL
1500
2000
2500
3000
TEMPERATURE, °C


°F
2500
3000
3500
4000
4500
5000
5400
TUNGSTEN - 3% RHENIUM (DOPED)
POWDER METALLURGY (DOPED)
PLASMA-FLAME SINGLE-CRYSTAL
ELONGATION, %
100
80
60
40
20
0
1500
2000
2500
3000
TEMPERATURE, °C


°F
2500
3000
3500
4000
4500
5000
5400
TUNGSTEN - 3% RHENIUM (DOPED)
POWDER METALLURGY (DOPED)
PLASMA-FLAME SINGLE-CRYSTAL
REDUCTION IN AREA, %
100
80
60
40
20
0
1500
2000
2500
3000
TEMPERATURE, °C


200 μ